Journal of Magnetism and Magnetic Materials 321 (2009) 3368–3372

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Review

Effect of Fe/Sr mole ratios on the formation and magnetic properties of $SrFe_{12}O_{19}$ microtubules prepared by sol–gel method

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ARTICLE INFO

Article history:
Received 17 January 2009
Received in revised form
3 April 2009
Available online 6 June 2009

Keywords:
$SrFe_{12}O_{19}$
Microtubule
Absorbent cotton template
Magnetic property

ABSTRACT

The sol was obtained by sol–gel method. Then, the sol was dripped onto the absorbent cotton template. The gel was obtained after the evaporation of water. Strontium ferrite microtubules were prepared after carrying out calcination process at different temperatures. The phase, morphology and particle diameter and the magnetic properties of samples were studied by X-ray diffraction (XRD), scanning electron microscopy (SEM) and vibrating sample magnetometer (VSM), respectively. The effects of Fe^{3+}/Sr^{2+} mole ratio and calcination temperature on the crystal structure, morphology and magnetic properties of ferrite microtubules were studied. The external diameters of obtained $SrFe_{12}O_{19}$ microtubules were found to range between 8 and 13 μm; the wall thicknesses ranged between 1 and 2 μm. When the Fe^{3+}/Sr^{2+} mole ratio and the calcination temperature were 11.5 and 850 °C, respectively, the coercivity, saturation magnetization and remanent magnetization for the samples were 7115.1 Oe, 70.1 and 42.4 emu/g, respectively. The mechanism of the formation and variation in magnetic properties of the microtubules were explained.

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M-type strontium hexaferrite ($SrFe_{12}O_{19}$) was discovered in the 1950s by Philips' laboratories [1]. Due to its appropriate magnetic properties, chemical stability and low cost compared with rare-earth compounds, it has attracted extensive interests in the past decades [2–4]. It is a hard magnet with high coercivity, which originates from high magneto-crystalline anisotropy with single easy magnetization axis. It has been recognized that it can be used as permanent magnets and recording media; it can also be used in telecommunication and as components in microwave, higher-frequency and magneto-optical devices [5]. The properties of this magnetic material are related to purity, size and morphology of the precursor powder [6,7]. By modifying its microstructure and controlling its chemical composition, size and morphology, its properties can be improved [8,9]. Therefore, the preparation of nano-$SrFe_{12}O_{19}$ having high purity, ultrafine size, good dispersion and excellent magnetism has been the focus of recent research [10–12]. Various preparation approaches and techniques, such as chemical coprecipitation [13], hydrothermal [14], sol–gel [15], glass crystallization [16], microemulsion [17], citrate precursor [18–20] and salt melt methods [21], have been developed; however, the preparation of $SrFe_{12}O_{19}$ microtubules has yet not been reported.

In this study, $Sr(NO_3)_0$, $Fe(NO_3)_3$ and citric acid were blended in a solution to obtain sol. Then the sol was dripped onto the absorbent cotton template. After the volatilization of the moisture, gel was formed on the surface of the absorbent cotton template. This gel was dried to obtain dry-gel. Then the dry-gel was calcined under different temperatures for 2 h in a muffle furnace to obtain $SrFe_{12}O_{19}$ microtubules. The phase of the samples was studied by X-ray diffraction (XRD), and the morphology and particle diameter of the samples were determined using scanning electron microscopy (SEM). The magnetic properties of samples were examined by using a vibrating sample magnetometer (VSM). The effect of Fe^{3+}/Sr^{2+} mole ratio on the synthesis of nanocrystalline strontium hexaferrite microtubules by sol–gel method was studied. Additionally, the formation of the strontium ferrites microtubules and the influence of Fe^{3+}/Sr^{2+} on the magnetic performance of the microtubules were explained.

1. Experiment

Weighted 0.833 g $Sr(NO_3)_2$ and $Fe(NO_3)_3 \cdot 9H_2O$ (as the mole ratios of Fe^{3+}/Sr^{2+} were 9, 10, 11, 11.5, 12 and 13) were dissolved in 200 ml de-ionized water. Then citric acid (weighed as the citric acid/metal ion mole ratio of 1:1) was added to the above mixture. The pH value was adjusted to 6 after the solution was agitated homogeneously. Subsequently, the solution was heated and agitated until the volume was approximately 140 ml to form sol. The sol was dripped onto the prepared loose and dry absorbent cotton fiber. Further, the dipped absorbent cotton was dried for 24 h at room temperature and then dried in a drying cabinet at

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doi:10.1016/j.jmmm.2009.05.066

70 °C. The dried gels were calcined under different temperatures (750, 850, 950 and 1050 °C) for 2 h to obtain $SrFe_{12}O_{19}$ microtubules.

The magnetic properties of the product were observed using a VSM at room temperature under a maximum field of 15 T. The reaction products were identified by XRD using Cu-Kα radiation. SEM was used to determine the grain size and morphology of the product.

2. Result and discussion

The morphology of $SrFe_{12}O_{19}$ was studied by SEM technology. Fig. 1 shows the SEM photographs of absorbent cotton and $SrFe_{12}O_{19}$ microtubules. Fig. 1A shows the SEM photographs of absorbent cotton. Fig. 1B–E shows the SEM photographs for the prepared samples with Fe^{3+}/Sr^{2+} mole ratios of 9, 10, 11.5, and 13, respectively, The samples were all calcined at 850 °C for 2 h. Fig. 1F and G shows the SEM photographs for the samples prepared at the calcination temperatures of 750 and 1050 °C, respectively, the Fe^{3+}/Sr^{2+} mole ratio of which were 11.5. As shown in the Fig. 1, the samples kept the morphology of cotton fiber. The external diameters of $SrFe_{12}O_{19}$ microtubules ranged between 8 and 13 μm, and the wall thicknesses ranged between 1 and 2 μm. From Fig. 1A–D, it can be concluded that the Fe^{3+}/Sr^{2+} mole ratio does not have a significant effect on the morphology of the $SrFe_{12}O_{19}$ microtubules. As shown in the Fig. 1C, E and F, it can be concluded that the samples kept the morphology of cotton fiber, despite the variation in the calcinations temperature from 750 to 1050 °C. However, with the increase in the calcinations temperature, the tube walls became more thick and looser.

In the sol formation process, the citric acid complex was formed as a result of the complex reaction between citric acid and metal ions. This complex dissolved in distilled water to form sol. When the solution of the citric acid complex is heated, the volatilization of solvent increased the viscosity of solution, thereby forcing the complex molecules to come together. Therefore, the unstable citric acid complex molecules, which did not contain the activated function of the condensation–polymerization reaction, underwent cross-linking through hydrogen bonds, and resulted in the formation of network structure. Then, when the anions and solvent embedded into the network structure, gel was formed [16–17]. Absorbent cotton was used as the template in our experiment. As the surface of the absorbent cotton was ragged, when the sol is dripped onto the surface of the absorbent cotton, the citric acid complex is absorbed first. On volatilization of the moisture content in air, this complex formed hydrogen bonds with the −OH on the surface of the absorbent cotton. On the continued evaporation of the moisture content, the excessive active groups of citric acid complex combined through hydrogen bonds to form annulus-like gel on the surface of the absorbent cotton template. The template was carbonized and then gasified through the calcinations process. The samples keep the morphology of cotton fiber. Finally, $SrFe_{12}O_{19}$ microtubules were obtained. On increasing the calcinations temperature, the calcinations process became fierce and there was an increase in the amount of released gas. Hence, the wallop of gas on the tube wall increases, and the tube wall becomes thicker, looser and lacunaris.

Figs. 2 and 3 show the XRD spectra of the $SrFe_{12}O_{19}$ samples prepared with different Fe^{3+}/Sr^{2+} mole ratios under a calcination temperature of 850 °C and the $SrFe_{12}O_{19}$ samples prepared under different calcinations temperatures with a Fe^{3+}/Sr^{2+} mole ratio of 11.5, respectively. In comparison to the values of analogy peaks corresponding to the crystal faces and the JCPDS card (card code: 24-1207), it can be concluded that when the calcinations temperature was 850 °C, for mole ratios higher than 11.5, Fe_2O_3

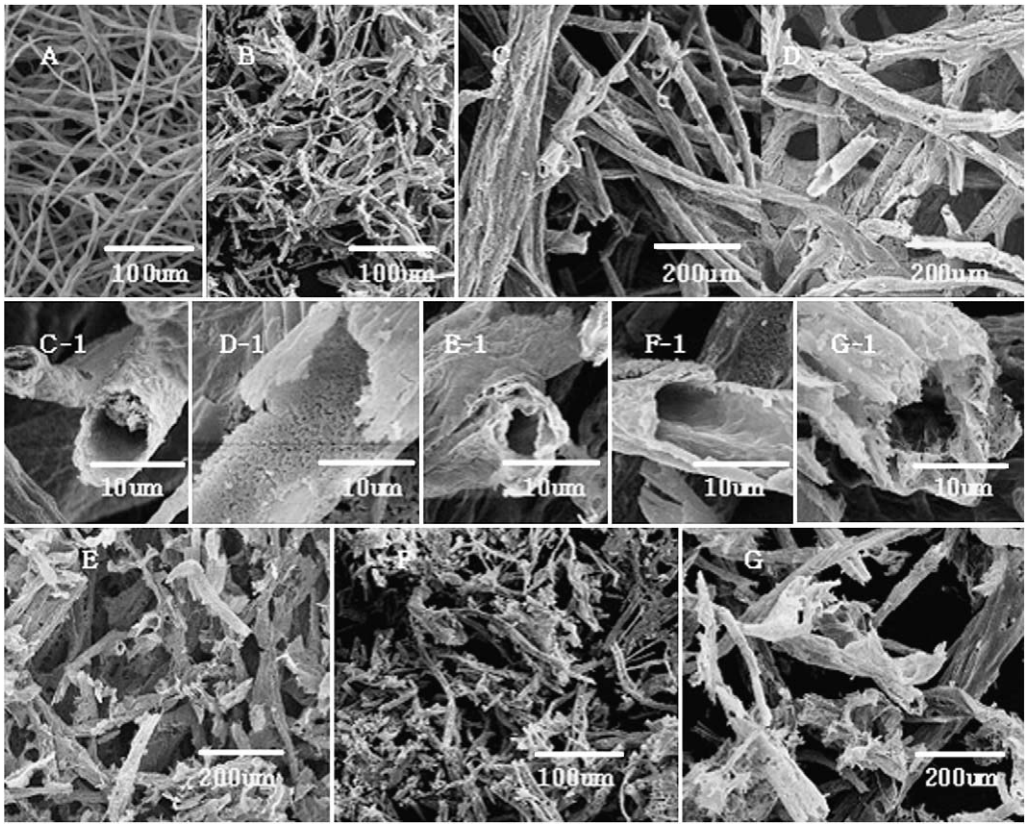

Fig. 1. SEM photographs of absorbent cotton and different $SrFe_{12}O_{19}$ microtubules.

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